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QUARTERLY PROGRESS REPORT 20

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

Prepared by
R. W. Harrison
J. P. Smith

Approved by
E. E. Hoffman

May 11, 1970

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-6474
R. L. Davies and P. L. Stone, Project Managers

NUCLEAR SYSTEMS PROGRAMS
SPACE SYSTEMS
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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CONTRACT NAS 3-6474

NASA Lewis Research Center
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Materials and Structures Division

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. R. L. Davies and P. L. Stone of NASA-Lewis Research Center are the NASA Technical Managers.

The program is being administered for the General Electric Company by E. E. Hoffman, and R. W. Harrison is acting as the Program Manager. Personnel making major contributions to the program during the current reporting period include:

T-111 Corrosion Loop Operation - A. Losekamp

Partial Pressure Gas Analysis - T. Lyon

1900°F Lithium Loop - J. Smith and T. Irwin

Advanced Tantalum Alloy Capsule Tests - G. Brandenburg

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from January 15, 1970 to April 15, 1970 of a three part program.

A. T-111 Rankine System Corrosion Test Loop

The primary task of this program is to fabricate, operate for 10,000 hours, and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation include the containment alloy, T-111 (Ta-8W-2Hf) and the turbine candidate materials Mo-TZC and Cb-132M which are located in the turbine simulator of the two-phase potassium circuit of the system. The loop design is similar to the Cb-1Zr Rankine System Corrosion Test Loop; a two-phase, forced convection, potassium corrosion test loop which was tested under Contract NAS 3-2547.[1] Lithium is heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop is accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

a. Boiling temperature, 2050°F
b. Superheat temperature, 2150°F
c. Condensing temperature, 1400°F
d. Subcooling temperature, 1000°F
e. Mass flow rate, 40 lb/hr
f. Boiler exit vapor velocity, 50 ft/sec
g. Average heat flux in plug (0-18 inches), 240,000 Btu/hr ft^2
h. Average heat flux in boiler (0-250 inches), 23,000 Btu/hr ft^2

[1] Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 7, NASA-CR-1509, 1970.

B. 1900°F Lithium Loop

Also included in the program is the fabrication, 7500-hour operation, and evaluation of a 1900°F, high flow velocity (1 gpm), pumped lithium loop designed to evaluate the compatibility of T-111 clad fuel specimens, ASTAR 811 type alloys, T-111, Mo-TZM and W-Re-Mo Alloy 256,* at conditions simulating a space power reactor system.

C. Advanced Tantalum Alloy Capsule Test

In addition to the primary program task cited above the program also includes capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Re-1Hf) in both potassium and lithium. Refluxing potassium capsule tests at 2200°F and lithium thermal convection capsule tests at 2400°F have completed 5000 hours of testing and the test specimens are being evaluated.

*W-25 a/o Re-30 a/o Mo (W-29 w/o Re-18 w/o Mo)

II. SUMMARY

On March 18, 1970 the T-111 Rankine System Corrosion Test Loop successfully completed the planned 10,000 hours of continuous operation. The alkali metal has been drained from the loop and preparation for posttest evaluation has been initiated. Visual examination of the loop showed it to be in excellent condition.

The 1900°F Lithium Loop achieved test conditions on January 31, 1970. The loop is operating satisfactorily and as of April 15, 1970, 1725 test hours have been accumulated.

The two ASTAR 811C potassium reflux capsules successfully completed 5000 hours of testing on February 24, 1970. One of the capsules has been opened and is being prepared for posttest evaluation.

III. PROGRAM STATUS

A. T-111 Rankine System Corrosion Test Loop

1. Loop Operating Temperatures

The T-111 Corrosion Test Loop successfully completed the planned 10,000 hours of continuous operation on March 18, 1970. The loop temperatures recorded just prior to the shutdown are shown in Figure 1 and the temperatures of major interest are summarized in the schematic in Figure 2. The performance of the loop was excellent and the power input to the lithium heater remained on automatic control for the final 4000 hours of the test with excellent temperature control being maintained. A comparison of the operating conditions at 1000 hours, 5000 hours and 10,000 hours shown in Table I further exemplifies the stability of the loop's performance.

2. Turbine Simulator Performance

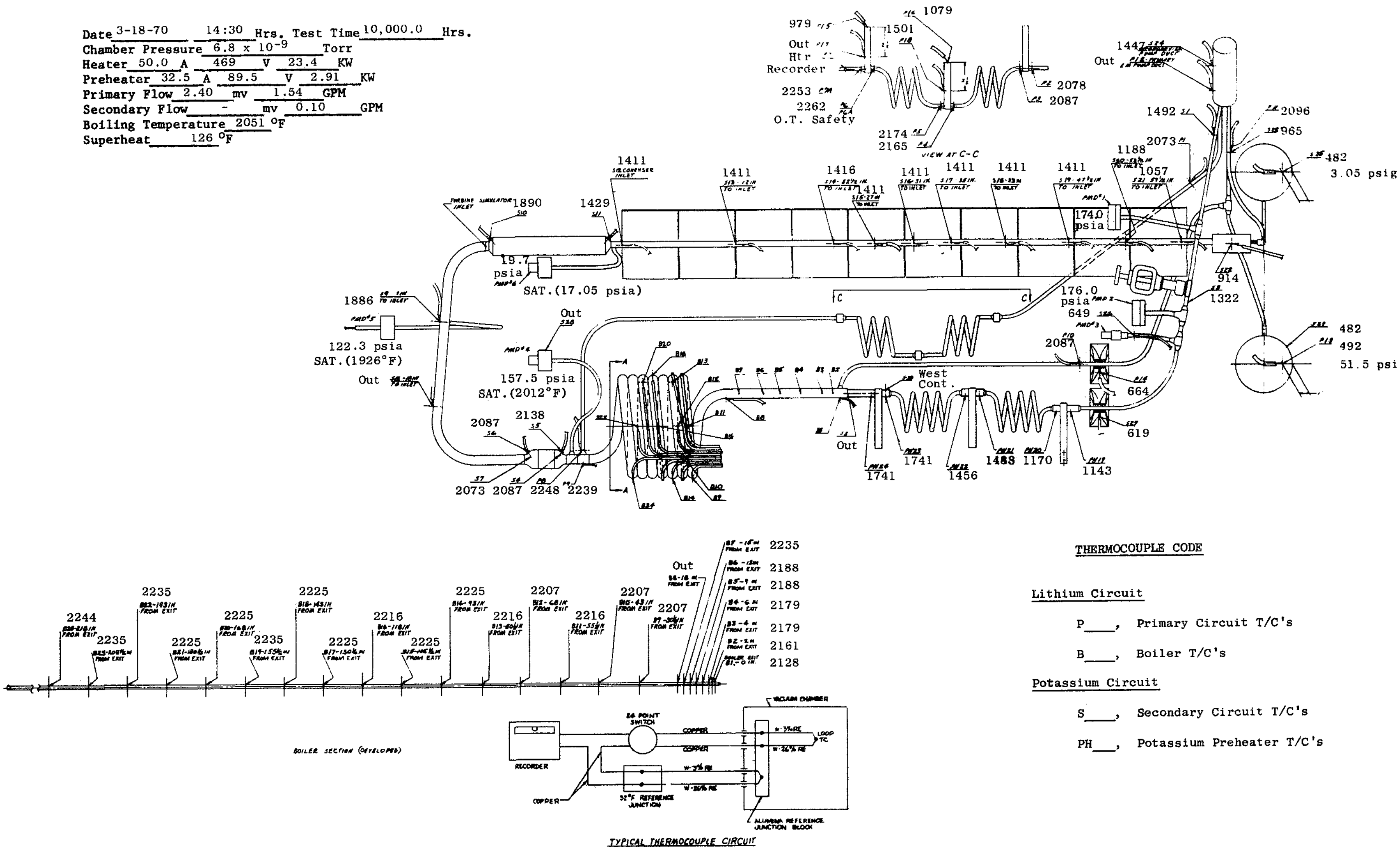
The calculated vapor velocities of the turbine simulator nozzles at 10,000 hours are presented in Table II for a mass flow rate of 36 lb/hr of potassium. The vapor velocity in the superheated first stage was 1030 ft/sec. The vapor velocity in the 88-percent-quality region ranged from a high of 1240 ft/sec in the second-stage nozzle to a low of 1095 ft/sec in the tenth stage nozzle. All vapor velocities were higher than the 1000 ft/sec design velocity.

The higher than design velocity is attributed to the lower than predicted vapor pressure at the inlet to the turbine simulator due to a higher than predicted pressure drop in the boiler. The higher than predicted pressure loss in the boiler is due to the high heat transfer rate in the 18-inch-long plug and the resulting high vapor quality in the entrance section of the boiler. For a given mass flow rate, the pressure drop in the tube is inversely proportional to the vapor density.

3. Test Chamber Environment-Partial Pressure Analysis

The chamber pressure and partial pressures of the various gas species in the test chamber during the entire 10,000 hours of loop operation are

Date 3-18-70 14:30 Hrs. Test Time 10,000.0 Hrs.
Chamber Pressure 6.8 x 10^{-9} Torr
Heater 50.0 A 469 V 23.4 KW
Preheater 32.5 A 89.5 V 2.91 KW
Primary Flow 2.40 mv 1.54 GPM
Secondary Flow - mv 0.10 GPM
Boiling Temperature 2051 °F
Superheat 126 °F



THERMOCOUPLE CODE

Lithium Circuit

P___, Primary Circuit T/C's

B___, Boiler T/C's

Potassium Circuit

S___, Secondary Circuit T/C's

PH___, Potassium Preheater T/C's

AS-885

Figure 1. T-111 Rankine System Corrosion Test Loop Thermocouple Instrumentation Layout.

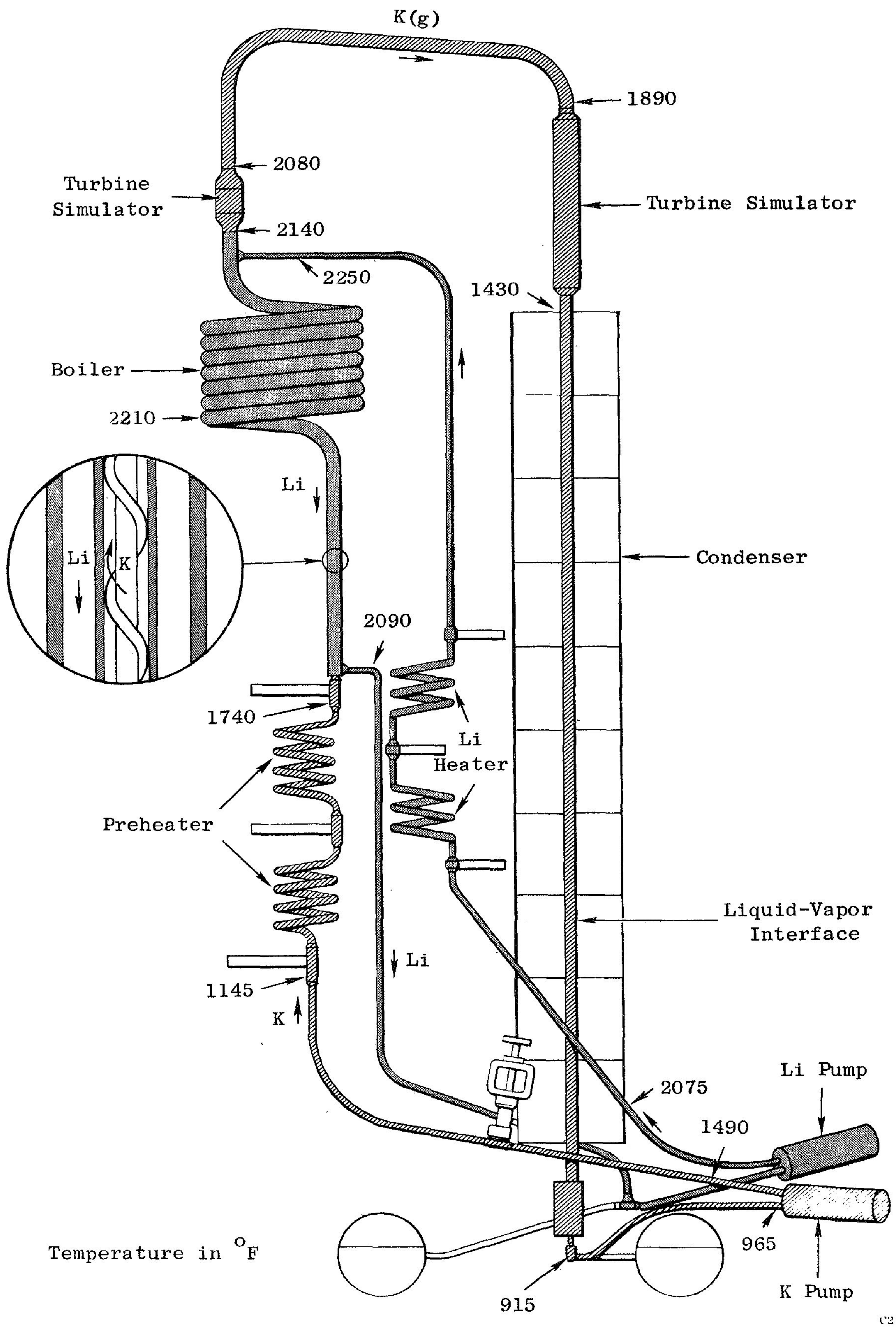


Figure 2. T-111 Corrosion Test Loop Operating Temperatures - 10,000 Hours.

TABLE I

T-111 RANKINE SYSTEM CORROSION TEST LOOP PERFORMANCE

Date	3-8-69	8-22-69	3-18-70
Test Hours	1000	5000	10,000
Lithium Flow Rate	205 lbs/hr	229 lbs/hr	234 lbs/hr
Lithium Temperature, In	2253 °F	2239 °F	2239 °F
Lithium Temperature, Out	2078 °F	2078 °F	2087 °F
Lithium ΔT	175 °F	161 °F	152 °F
Potassium Flow Rate	36 lbs/hr	38 lbs/hr	36 lbs/hr
Plug Boiling Temperature	2048 °F	2052 °F	2050 °F
Boiler Exit Vapor Temp.	2147 °F	2137 °F	2138 °F
Boiler Exit Saturation Temp.	2012 °F	2012 °F	2012 °F
Potassium Vapor Superheat	135 °F	125 °F	126 °F
Condensing Temperature	1416 °F	1411 °F	1411 °F
Potassium Heat Input			
1. Preheat	2280 Btu/hr	2410 Btu/hr	2260 Btu/hr
2. Heat of Vaporization	26,300 Btu/hr	27,600 Btu/hr	26,500 Btu/hr
3. Superheat	1040 Btu/hr	960 Btu/hr	920 Btu/hr
TOTAL	29,620 Btu/hr	30,970 Btu/hr	29,680 Btu/hr
Total Power to Lithium Heater	13.2 kw	13.8 kw	14.0 kw
Total Power to Potassium	8.7 kw	9.0 kw	8.7 kw
Net Heat Loss	4.5 kw	4.8 kw	5.3 kw

TABLE II

T-111 RANKINE SYSTEM CORROSION TEST LOOP TURBINE SIMULATOR PERFORMANCE AT 10,000 HOURS

Nozzle Number	Material	Nozzle Diameter (R.T.), Inch	Inlet Temperature °F	Exit Pressure PSIA	Vapor Velocity* ft/sec
1	Mo-TZC	0.0892	2142	110.0	1030
2	Mo-TZC	0.0881	1890	91.0	1240
3	Mo-TZC	0.0964	1834	74.0	1245
4	Mo-TZC	0.1083	1774	60.0	1200
5	Mo-TZC	0.1181	1716	49.5	1205
6	Cb-132M	0.1292	1664	40.0	1220
7	Mo-TZC	0.1457	1611	32.5	1165
8	Mo-TZC	0.1598	1562	26.3	1180
9	Cb-132M	0.1784	1514	21.5	1140
10	Mo-TZC	0.1986	1471	17.6	1095

*Nozzle exit

summarized in Figure 3. The data for the initial 1000 hours of testing are shown on an expanded scale since this is where the largest changes were observed. Following this initial large decrease, there was a continued gradual decrease in most species for the period 1000 to 6500 hours at which time all pressures essentially leveled off and remained constant until the shutdown at 10,000 hours. Only a limited number of data points are shown for clarity; however, residual gas analysis were obtained at least every 24 hours.

4. Termination of the Loop Test

The loop test was terminated at 1430 hours on March 18, 1970 without the loss of a day in the test period. Only four one-hour interruptions in the testing were experienced during the 10,000 hour test, and they resulted from power interruptions to the test facility and not from loop performance. The continuous operation of the T-111 Rankine System Corrosion Test Loop can be considered a major milestone in the development of technology for advanced space power systems.

The loop shutdown was performed in gradual steps to reduce the possibility of large thermal shocks from boiling instabilities. The plot of the pressures on either side of the metering valve, Figure 4, best describes the loop shutdown procedure and the small instabilities which were observed when the potassium temperature was reduced below 1600°F. At each step, the power to the lithium heater, potassium preheater, and both EM pumps and the potassium surge tank pressure was reduced. The surge tank pressure was finally reduced to 0 psia and the potassium dumped into the surge tank.

5. Alkali Metal Sampling and Analysis

A sample of the hot potassium (1000°F) was obtained through the sampling system shown schematically in Figure 5. The potassium remaining in the surge tank was then immediately drained into the potassium still pot by gravity. This potassium was subsequently vacuum distilled off at 800°F. The pot was opened and a small amount (19 mg) of a black powdery residue was obtained for analysis. The analysis of the potassium sample and the black residue are shown in Table III. No major changes to the potassium were observed as a result of the 10,000 hour exposure to T-111 at a maximum temperature of 2150°F. The black residue

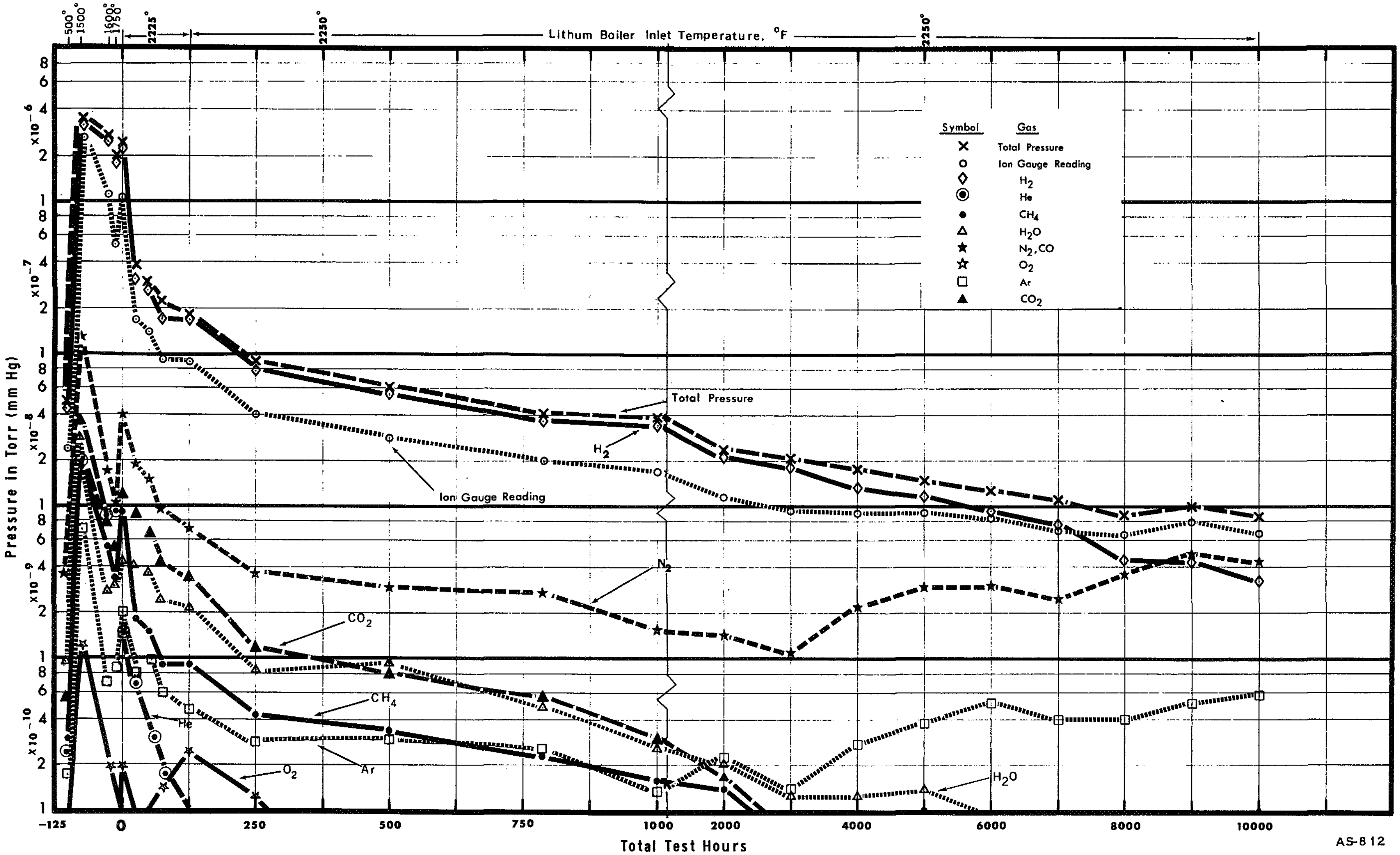


Figure 3. Test Chamber Environment During Testing of the T-111 Rankine System Corrosion Test Loop.

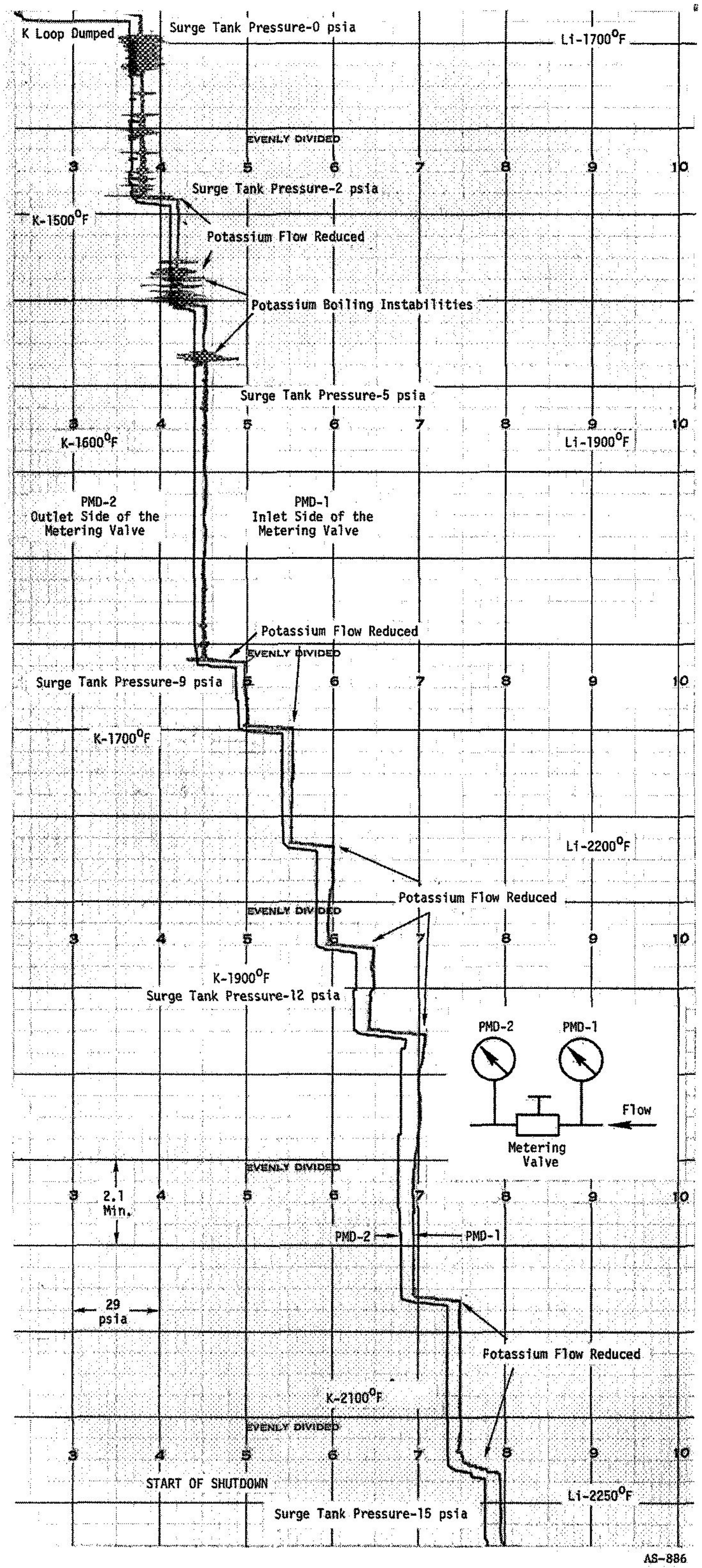


Figure 4. Potassium Pressure on Either Side of the Metering Valve During the Shutdown Completing the 10,000 Hours of Testing of the T-111 Rankine System Corrosion Test Loop.

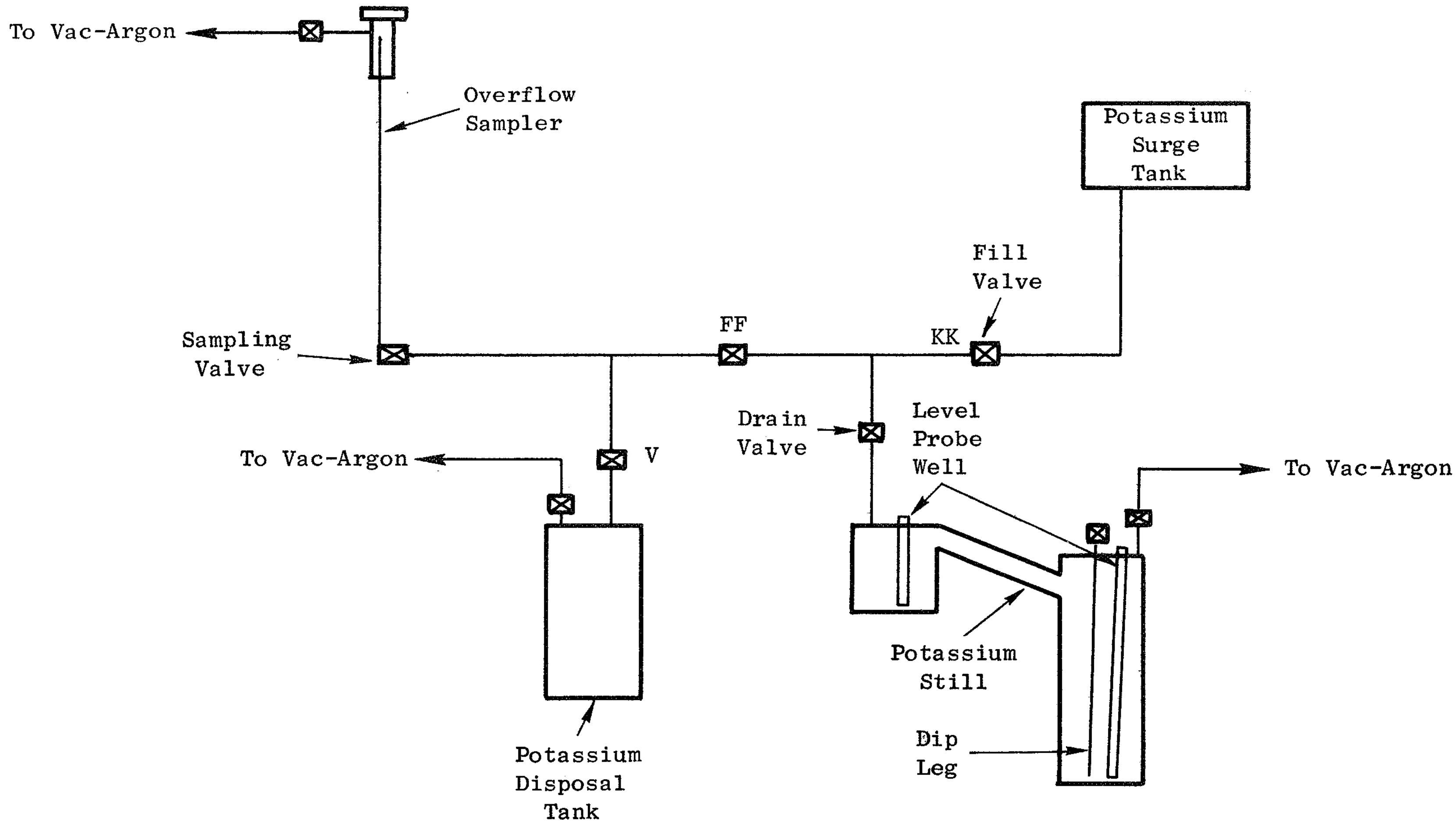


Figure 5. T-111 Corrosion Test Loop Potassium Draining and Sampling Schematic.

TABLE III

ALKALI METAL ANALYSES - T-111 RANKINE SYSTEM CORROSION TEST LOOP

	Potassium		Lithium		
	Pretest ppm	After 10,000 Hours ppm	Pretest ppm	After 10,000 Hours ppm	ppm
O	3,4	6,11	49	460,554	386,413
C	32	41	31	81	65
N	-	-	38,47	48,53	44,48
Ag	< 2	< 2	< 5	< 5	< 5
Al	10	2	5	5	< 5
B	< 30	< 30	< 50	< 75	< 75
Ba	< 20	< 10	< 50	< 75	< 75
Be	< 2	< 2	< 5	< 5	< 5
Ca	10	2	5	5	5
Cb	< 10	< 10	< 25	< 25	< 25
Co	< 2	< 2	< 5	< 5	< 5
Cr	< 2	< 2	< 5	< 5	< 5
Cu	2	< 2	5	5	5
Fe	2	< 2	< 5	< 5	< 5
Mg	2	< 2	5	5	5
Mn	< 2	< 2	< 5	< 5	< 5
Mo	< 2	< 2	< 5	< 5	< 5
Na	< 20	< 30	< 50	< 125	< 125
Ni	< 2	< 2	5	< 5	< 5
Pb	< 20	< 20	< 50	< 50	< 50
Si	2	2	5	5	5
Sr	< 2	< 2	5	5	5
Sn	< 10	< 10	< 25	< 25	< 25
Ti	< 10	< 10	< 25	< 25	< 25
V	< 10	< 10	< 25	< 25	< 25
Zr	< 10	< 10	< 25	< 25	< 25
Li	31	-	K = 106		

Black Residue After 10,000 Hour Test

Major (10 - 100%): Cr, Fe
Minor (1 - 10%): Ni, Ti, Li
Trace (0 - 1%): Ag, Al, Cu, Mg, Mn, Mo, Si, V

contained the elements of stainless steel plus lithium which were also observed after draining the original potassium charge.[2]

The initial lithium draining and sampling system was identical to that for potassium as shown previously in Figure 5; however, failure of the bellows in the original fill valve, required replacement of this valve, and the modified sampling system, shown in Figure 6, was installed. The lithium was hot (1100°F) dumped into the surge tank, and 300 cc of lithium were subsequently drained into the waste tank and a sample was obtained between the dump valve and the fill valve. This sample was removed and the fill and dump valves were cleaned. The in-line sample tube, shown by the dotted line in Figure 6, was then installed. Following leak checking and bakeout of the system the remaining lithium in the surge tank was drained into the waste tank at 1000°F thereby obtaining a second lithium sample in the transfer line. The lithium samples were analyzed and the results are presented in Table III. The results indicate that the oxygen concentration in the lithium increased about ten-fold during the test which could be expected as lithium would reduce the oxygen concentration of the T-111 piping (without corrosion).

6. Preparation for Posttest Evaluation

Following the removal of all alkali metal from the loop and surge tanks, all electrical power and water cooling was turned off, the loop and vacuum chamber were allowed to cool to room temperature, and the bell jar removed. Visual inspection of the loop and all related components showed them to be in excellent condition. Except for certain isolated areas, there was no discoloration of the Cb-1Zr dimpled foil thermal insulation. The few areas which showed discoloration were in close proximity to the stainless steel support structure. Photographs of the loop prior to any disassembly or removal of foil are shown in Figures 7, 8, and 9. Figure 7 shows the boiler (insulation package still in place), lithium inlet line to boiler, both turbine simulators, cross-over line, the top of the condenser section, and two pressure

[2] Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 12, for Period Ending June 14, 1968, NASA Contract NAS 3-6474, NASA-CR-72452, GESP-144.

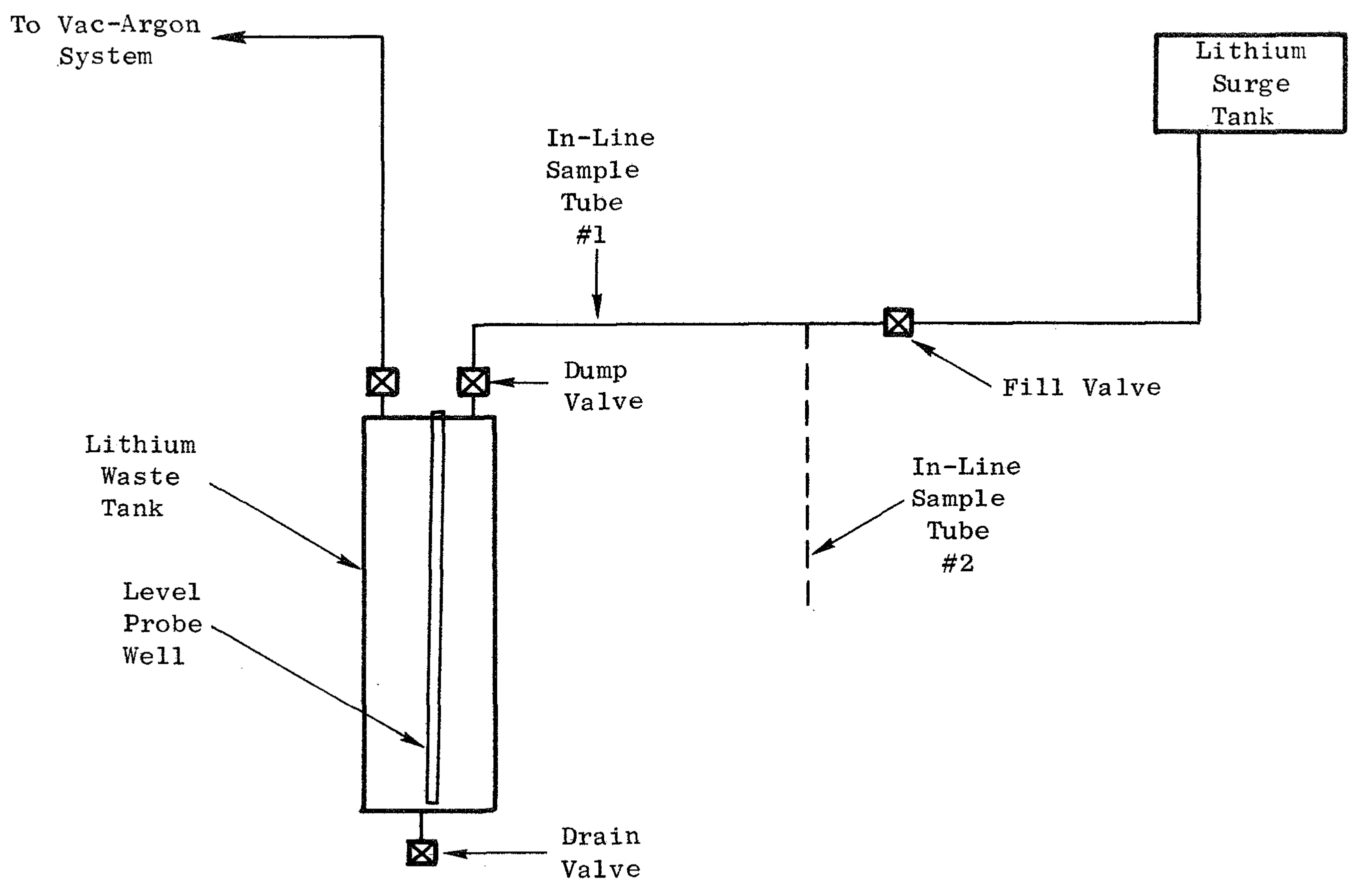


Figure 6. T-111 Corrosion Test Loop Lithium Draining and Sampling Schematic.

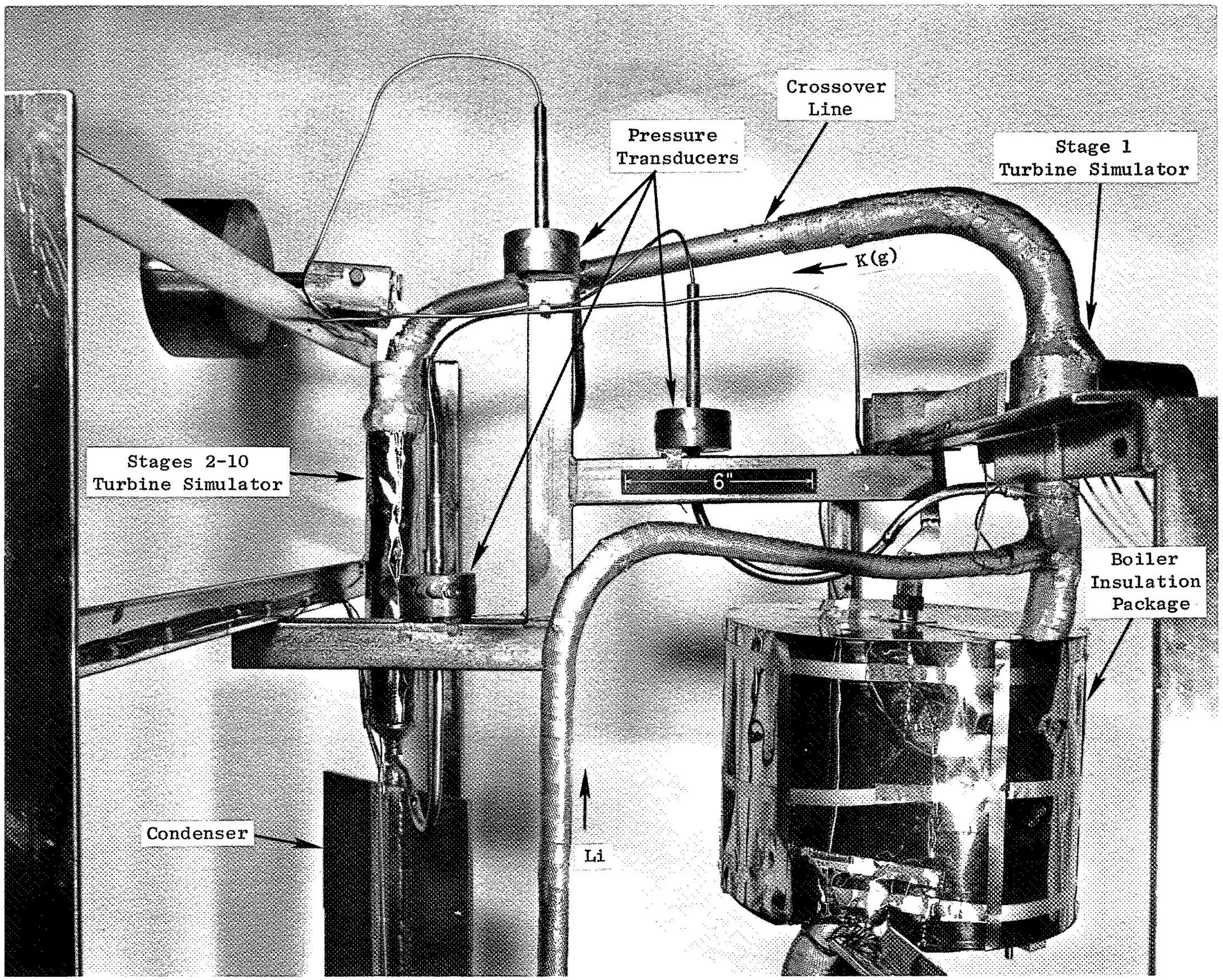


Figure 7. Top Portion of T-111 Corrosion Test Loop After Completion of 10,000 Hours of Continuous Testing. (P70-3-22G)

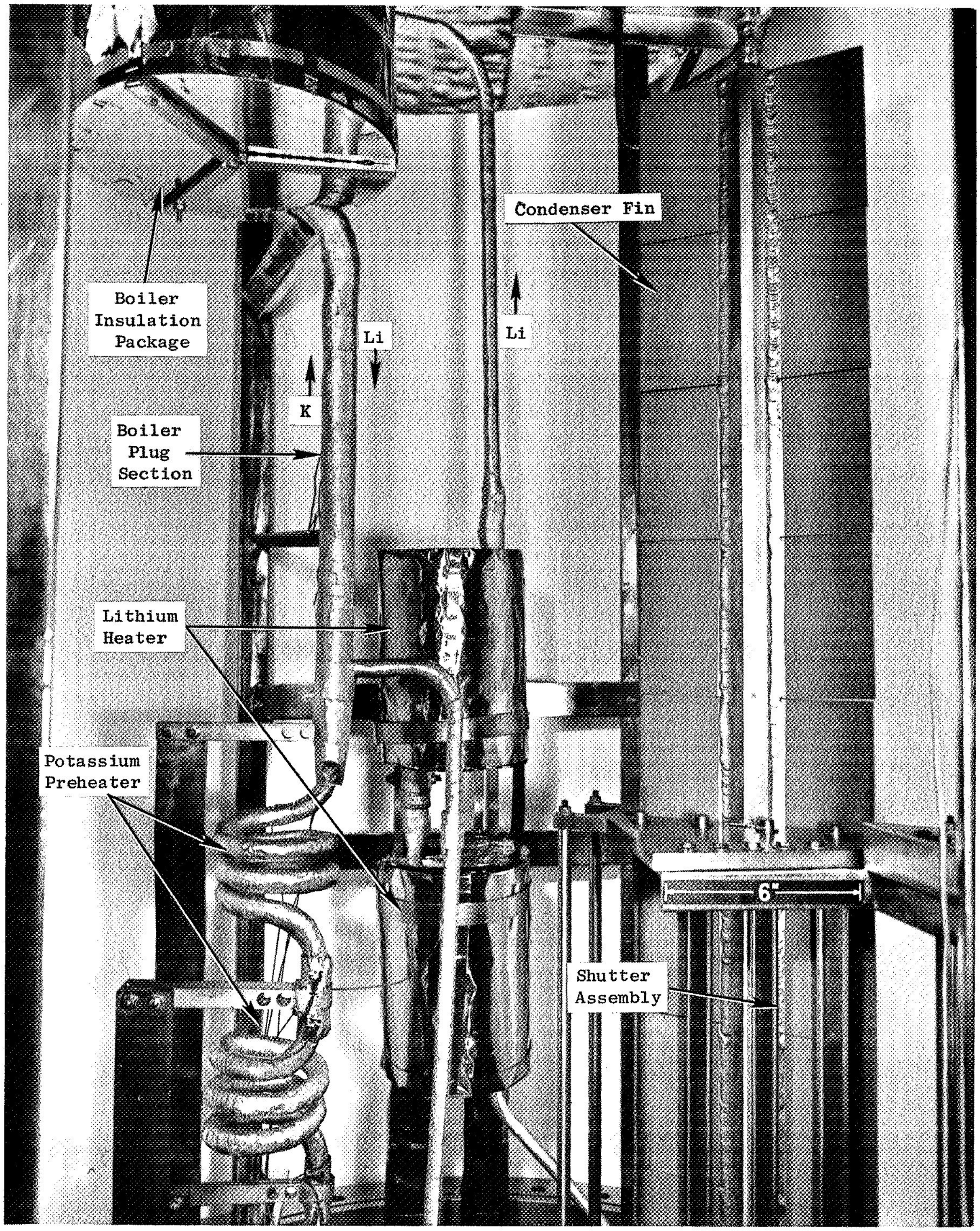


Figure 8. T-111 Corrosion Test Loop After Completion of 10,000 Hours of Testing. (P70-3-22C)

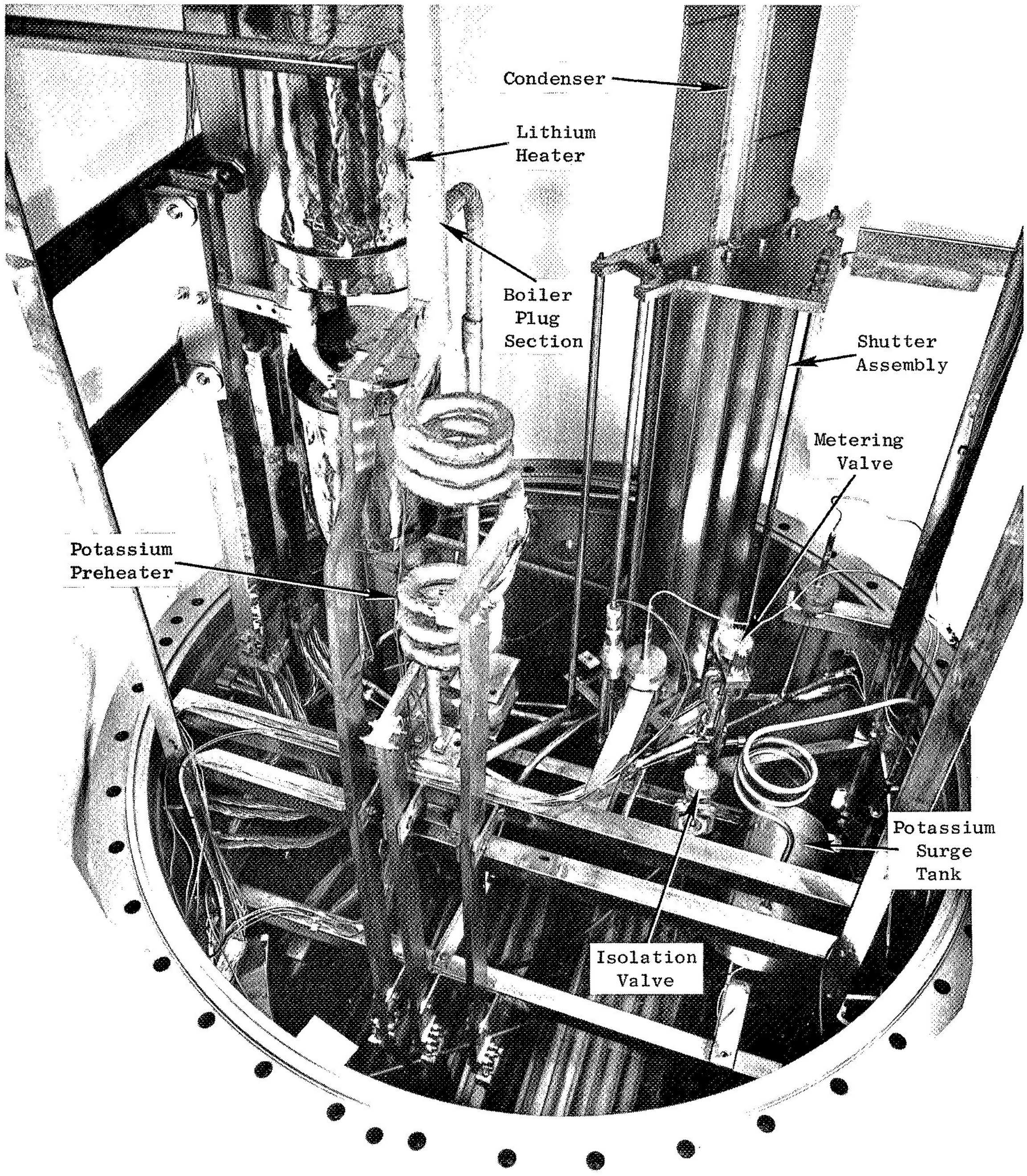


Figure 9. Lower Portion of T-111 Corrosion Test Loop After Completion of 10,000 Hours of Continuous Testing. (P70-3-22F)

transducers. Figure 8 shows the bottom of the boiler insulation package, boiler plug section, condenser, potassium preheater, lithium heater, and lithium inlet and return lines. Figure 9 is a photograph looking down into the spool piece illustrating, in addition to the items described above, the shutter assembly, potassium surge tank and both valve assemblies. Close examination of the metering valve revealed that the reason it became inoperative during the test is that the coupling connecting the spur gear shaft with the flexible cable had become disengaged. Following this initial visual examination, thermocouples and the thermal insulation were removed from the boiler, turbine simulators, and crossover line, the loop components which were to undergo posttest evaluation. The thermal insulation foil was kept sorted by layer and loop component for posttest chemical analysis. All thermal insulation was ductile and no evidence of contamination was observed. After removal of the thermal insulation, these components were reinspected visually. Again these components were found to be in excellent condition as shown in the photographs of the boiler and plug regions in Figures 10 and 11.

Auxiliary supports were installed as needed to support the loop during removal of the components. The techniques employed were similar to those developed during removal of the boiler.[3] The protective vinyl chamber was placed over the loop, sealed to the spool piece, evacuated and back filled with helium in preparation for cutting and removal of the desired components. The loop enclosed in the vinyl chamber and ready for cutting is shown in Figure 12. After filling the bag with helium, areas to be cut were first hand filed as necessary to remove weld metal build up or to reduce the wall thickness sufficiently to be finally cut with a tubing cutter. All final cutting was performed with a tubing cutter, at the areas shown in Figure 13, to eliminate the possibility of introducing chips inside the loop tubing. After making the cuts, both the potassium and lithium circuits were pressurized with argon to verify that no blockage occurred. After verification of the gas path, the open tube ends were plugged or capped with stainless steel "Swag-lok" fittings.

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[3] Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 13 for Period Ending July 15, 1968, NASA Contract NAS 3-6474, NASA-CR-72483, GESP-182.

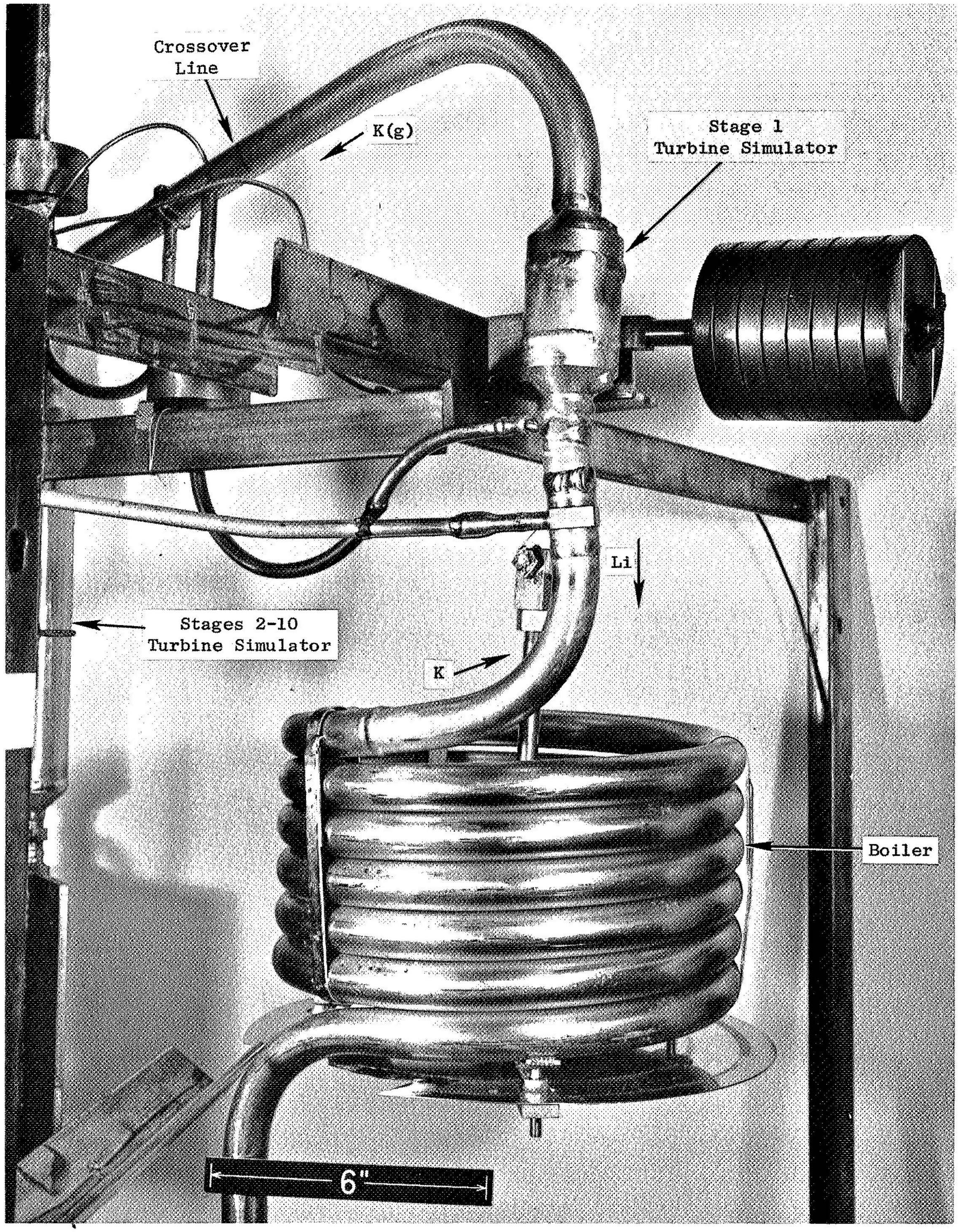

Figure 10. T-111 Corrosion Test Loop Boiler, and Turbine Simulator After 10,000 Hours of Continuous Operation. (P70-4-1A)

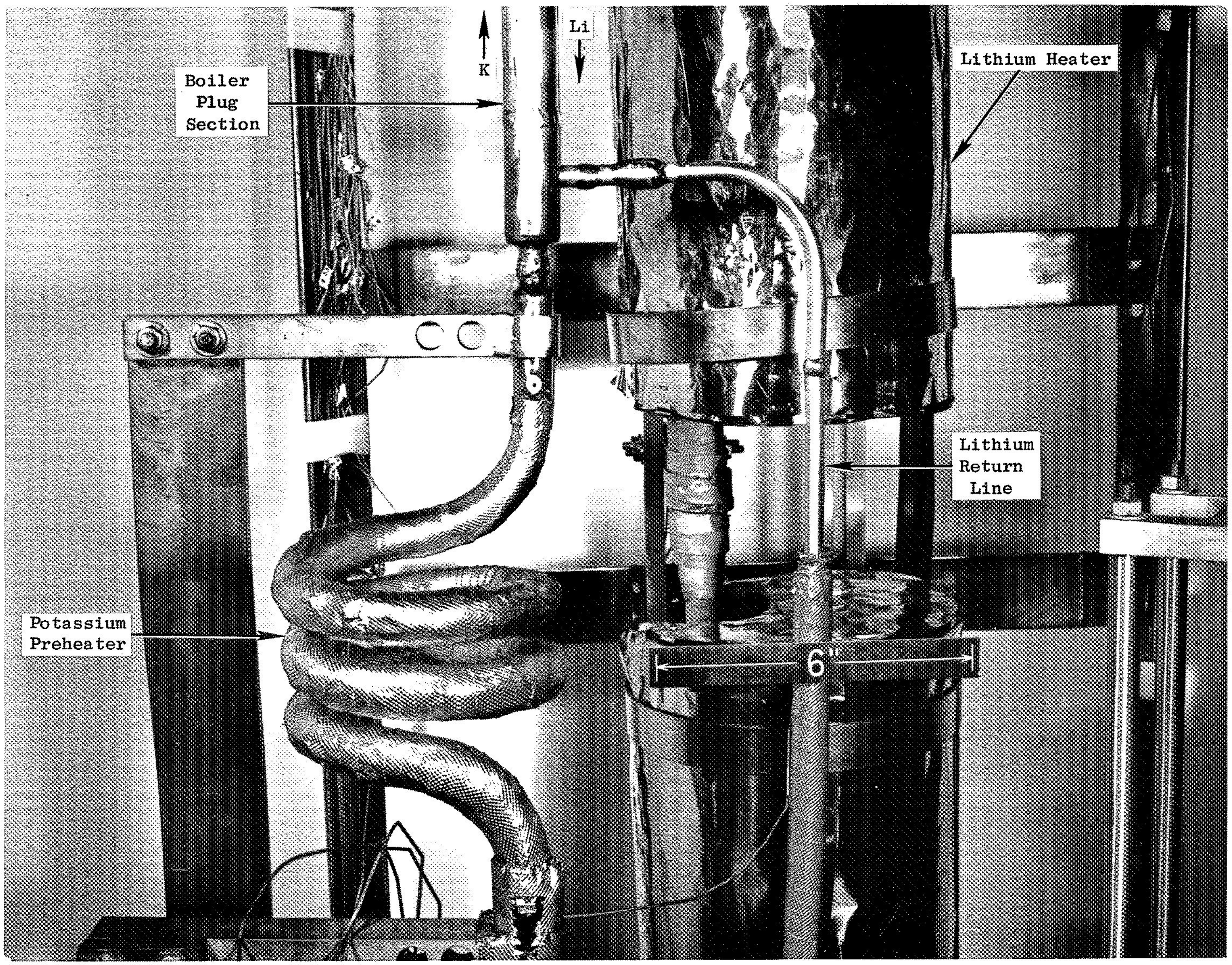

Figure 11. Components of T-111 Corrosion Loop After Removal of Thermal Insulation from Areas to Undergo Posttest Evaluation. (P70-4-1C)

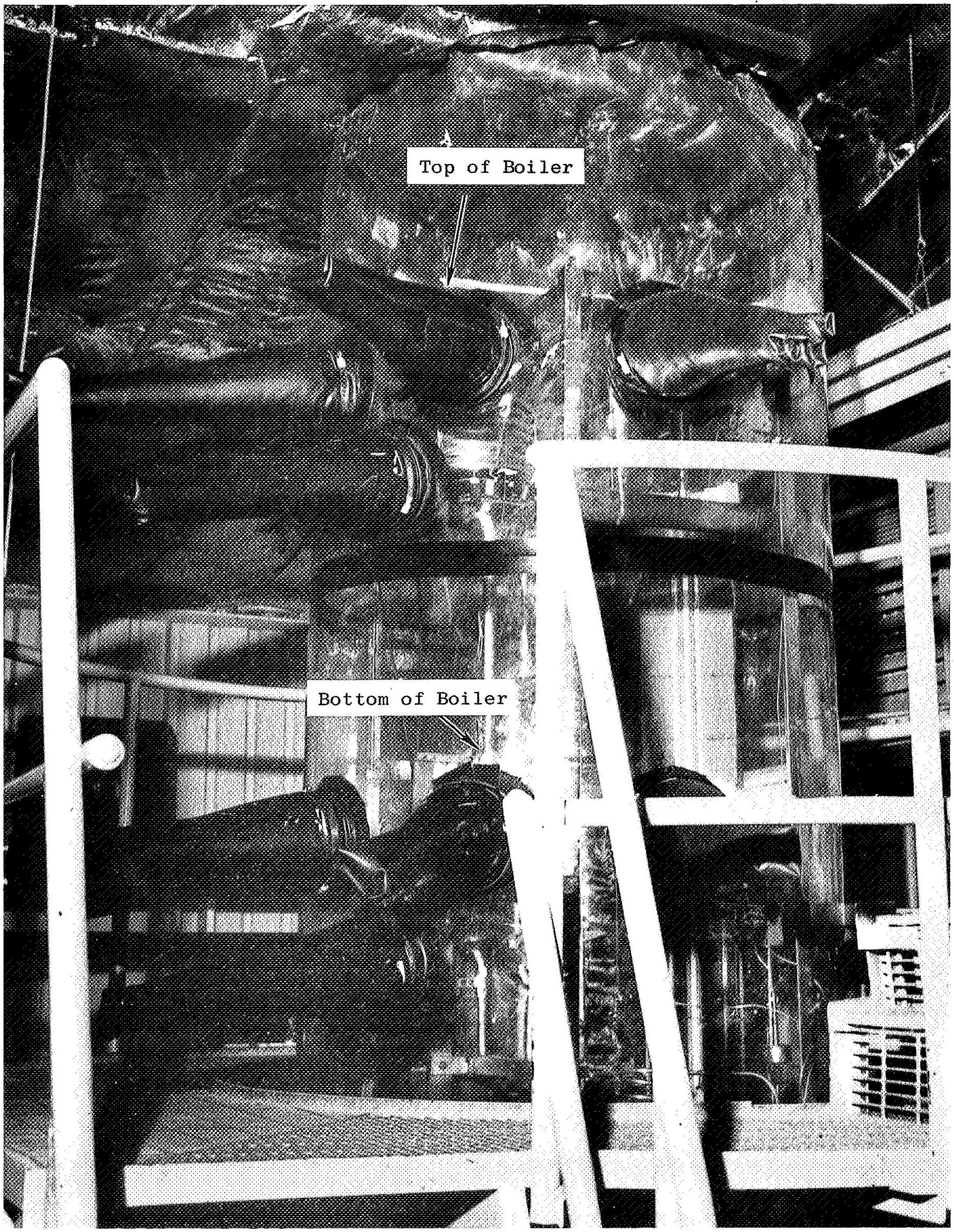

Figure 12. Vinyl Chamber Used for Cutting Operations on T-111 Corrosion Test Loop. (C68062842)

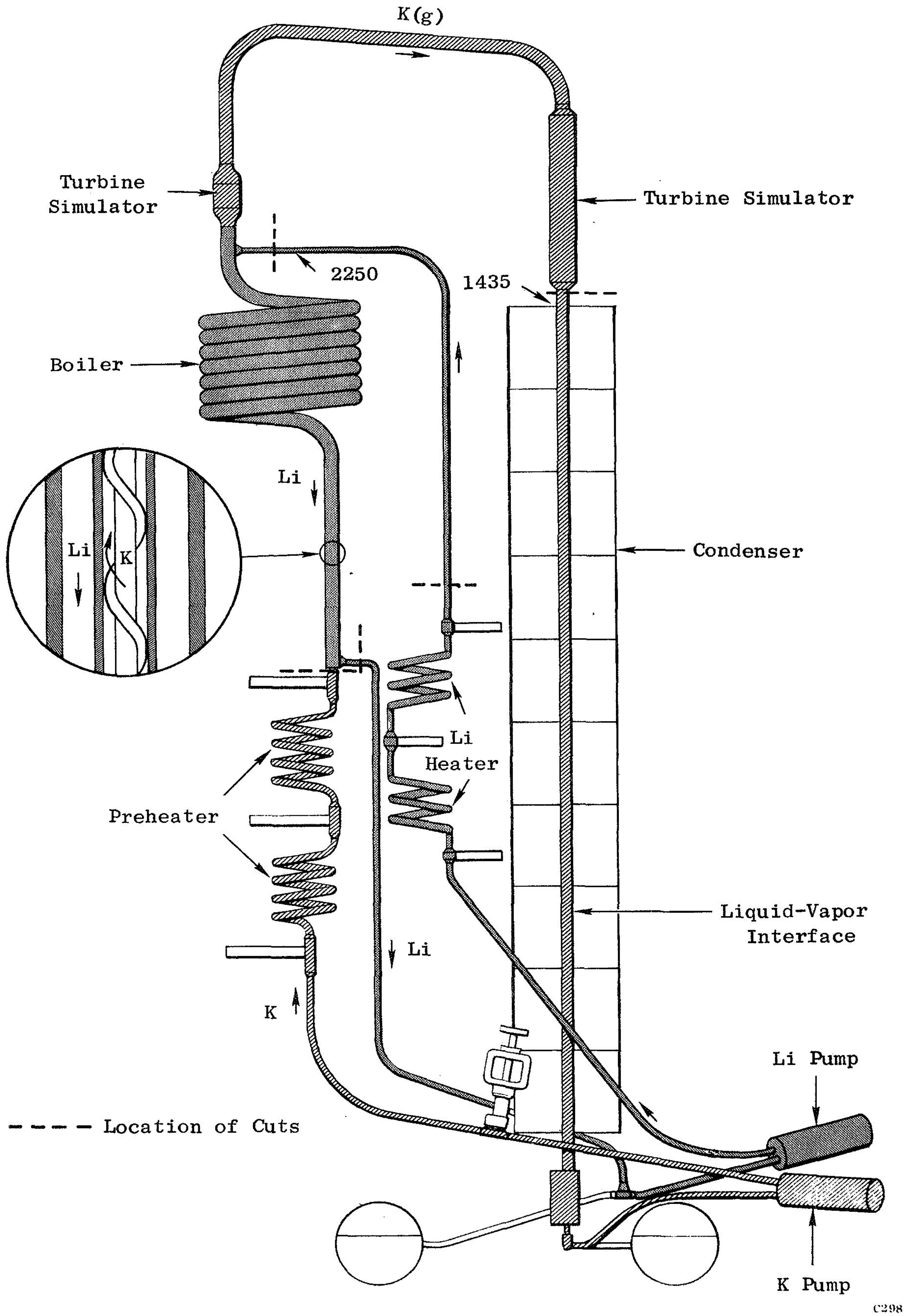

Figure 13. T-111 Corrosion Test Loop Showing Location of Cuts Necessary to Remove Boiler and Turbine Simulators for Evaluation at the Completion of 10,000 Hours of Testing.

The vinyl chamber was then removed, and the boiler and turbine simulators were removed from the chamber and placed on a lab bench for closer visual examination. The bell jar was replaced and the chamber evacuated to maintain the remainder of the loop under vacuum until needed in the future.

The boiler and turbine simulators after removal from the loop are shown in Figure 14. This assembly was then transferred to the weld chamber in Building 700 for attachment of valves to the potassium and lithium inlet tubing to facilitate removal of residual alkali metals with liquid ammonia.

B. 1900°F Lithium Loop

During the current reporting period; checkout and calibration of all safety, control and instrumentation circuits of the 1900°F Lithium Loop was completed; and on January 31, 1970 the loop achieved the desired test conditions. On April 15, 1970 the loop had completed 1725 hours of testing.

1. Loop Operation

The loop was operated on manual heater temperature control for the first 100 hours. Since only minor power adjustments were required during this initial 100 hours of operation, the heater power was switched to automatic control and has been operating in this mode for the entire test with less than 10°F temperature variation. During this period the loop has experienced several shutdowns, totaling 55 hours, resulting from safety circuits being activated. The major cause of the shutdowns was the result of voltage instabilities caused by inclement weather. None of the shutdowns were caused by loop instabilities. Two steps were completed to minimize shutdowns in the future. First, an alarm system was connected with the GE-NSP Security Headquarters and is activated when the loop temperature exceeds preset boundaries during non-regular working hours. Secondly, a 440 volt, 100 kva constant voltage transformer was connected to the main power supply. Since the installation of the voltage stabilizer, no shutdowns have been incurred.

2. Test Chamber Environment - Partial Pressure Analysis

The chamber pressure and partial pressure of the various gaseous

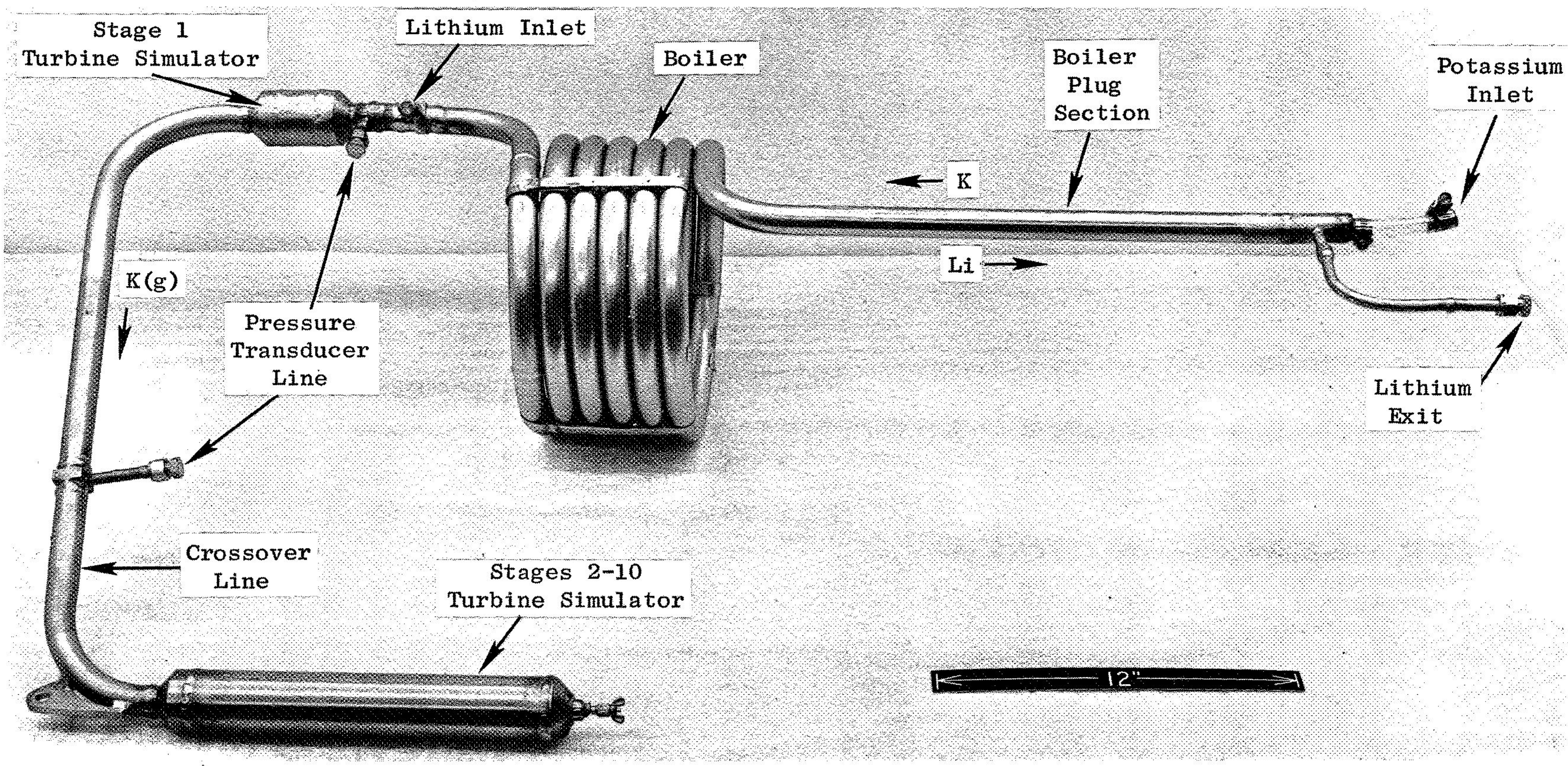

Figure 14. Boiler, Crossover Line and Turbine Simulators After Removed From T-111 Corrosion Test Loop. (P70-4-14A)

species in the test chamber during the current period are shown in Figure 15. During the initial 100 hours all pressures dropped rather sharply; this was followed by a somewhat decreased rate of pressure drop during the next 650 hours, and during the period 750-1750 hours there is a general trend for the partial pressure of most gaseous species to be leveling off. Data points are shown only at 250 hour intervals for clarity; however, residual gas analysis are obtained at least every 24 hours; and the ion gage pressure is monitored continuously. This data for the 1900°F Lithium Loop is in good agreement with the T-111 loop data presented earlier (Figure 3) in this report in that H_2, N_2-CO, and Ar are the major gas species present in the chamber.

3. Loop Operating Temperatures

A summary of all loop temperatures is shown in Figure 16. Temperature distribution is excellent, and the 100°F temperature drop across the corrosion specimen section is as predicted. The defective thermocouples are primarily the result of thermal cycles caused by the shutdowns discussed earlier; these thermocouples will be repaired at the planned 2500 hour shutdown.

C. Advanced Tantalum Alloy Capsule Tests

The two ASTAR 811C potassium reflux capsules completed 5000 hours of testing on February 24, 1970. Posttest visual examination revealed nothing of an unusual nature except for slight thermally etched areas and a gold colored deposit at the bottom of each capsule. These observations are similar to what was observed on the lithium thermal convection capsules run previously. Condensing rates and operating conditions for the reflux capsules just prior to completion of the test are summarized in Table IV.

A complete posttest evaluation will be performed on capsule No. 2 since it showed a higher reflux rate than capsule No. 1 for the 5000 hour test. Capsule No. 1 will be retained intact for possible further testing. Capsule No. 2 has been opened, cleaned and the specimens removed. Posttest evaluation of the capsule and specimens is underway.

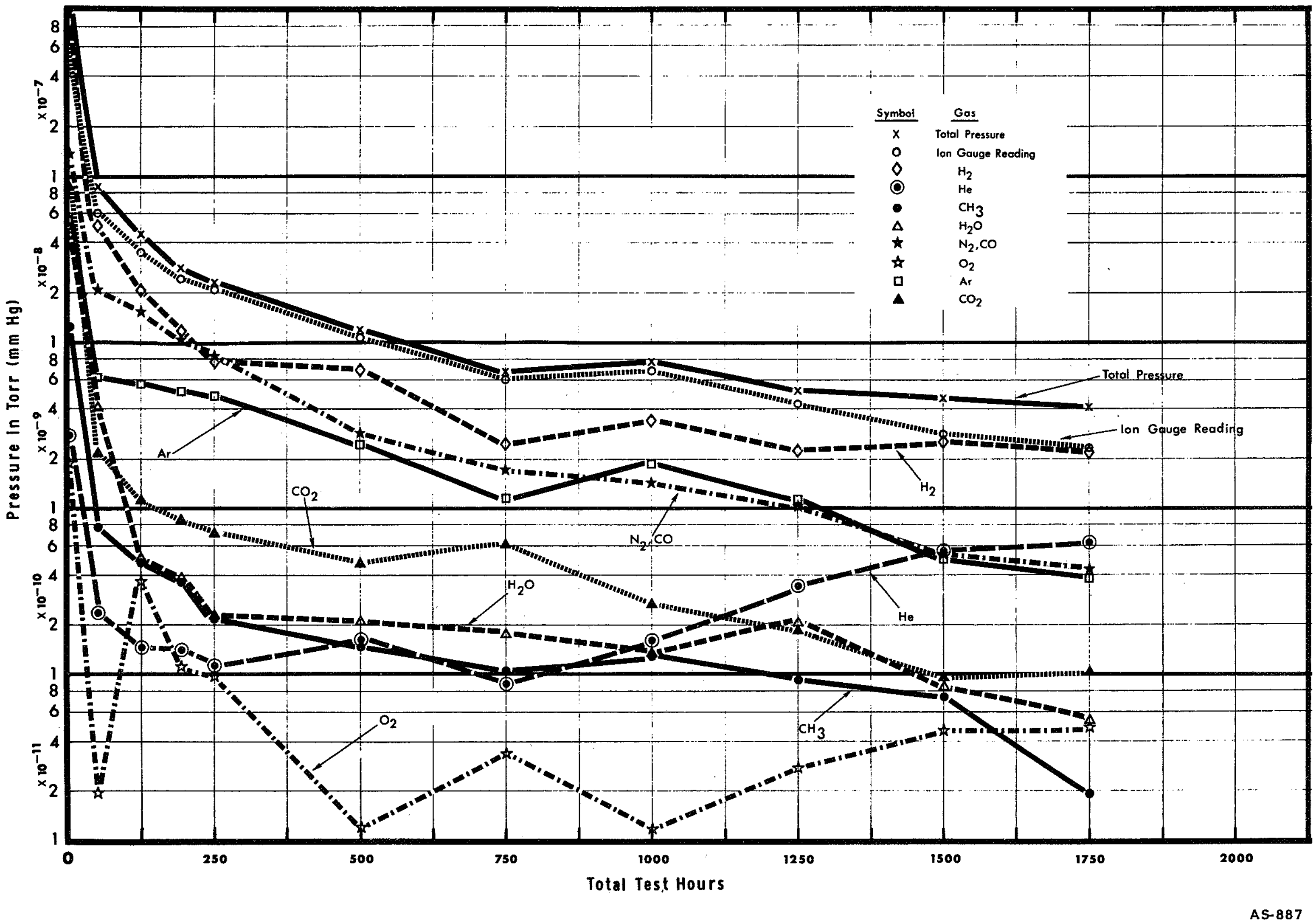

Figure 15. Test Chamber Environment During Testing 1900°F Lithium Loop.

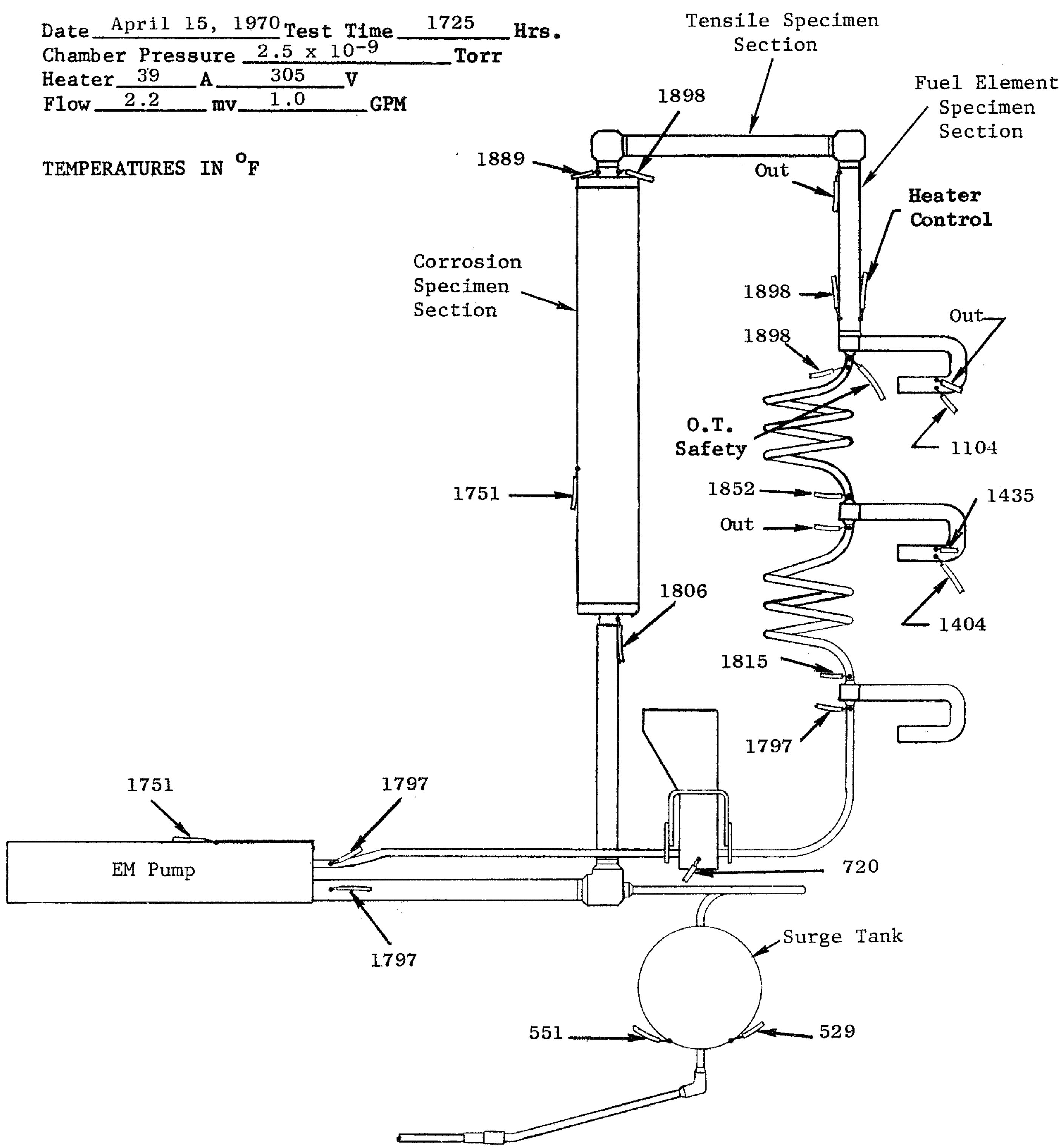

Figure 16. 1900°F Lithium Loop Operating Temperatures - 1725 Hours

TABLE IV

CONDENSING RATES AND OPERATION CONDITIONS FOR ASTAR 811C POTASSIUM REFLUX CAPSULES

Capsule 1	
Average Condensing Temperature	2194°F
Condensing Rate at Average Temperature	30.9 ± 0.30 lbs/hr ft^{2}*
Average Power Input	1567 watts
Average Power Loss	59.9%
Vacuum	6.3 x 10^{-9} torr
Total Test Hours	5003 hours
Capsule 2	
Average Condensing Temperature	2177°F
Condensing Rate at Average Temperature	33.0 ± 0.27 lbs/hr ft^{2}*
Average Power Input	1517 watts
Average Power Loss	55.9%
Vacuum	6.3 x 10^{-9} torr
Total Test Hours	5003 hours

* Limits represent 95% confidence level based on least squares linear analysis.

IV. FUTURE PLANS

Continue posttest evaluation of the T-111 Rankine System Corrosion Test Loop.

Complete the planned initial 2500 hours of testing on the 1900°F Lithium Loop, remove two of the three T-111 clad fuel element specimens, and replace these specimens with two others; one to have a purposely defective clad.

Complete posttest evaluation of the ASTAR 811C potassium reflux capsule and specimens.

PREVIOUSLY PUBLISHED PROGRESS REPORTS FOR THIS CONTRACT

Quarterly Progress	For Quarter Ending
Report No. 1 (NASA-CR-54477)	July 15, 1965
Report No. 2 (NASA-CR-54845)	October 15, 1965
Report No. 3 (NASA-CR-54911)	January 15, 1966
Report No. 4 (NASA-CR-72029)	April 15, 1966
Report No. 5 (NASA-CR-72057)	July 15, 1966
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